भारतीय मानक

Indian Standard

IS 8494 : 2023

एम.सी.पी.ए., तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

MCPA, Technical – Specification

(First Revision)

ICS 65.100.20

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

May 2023 **Price Group 5**

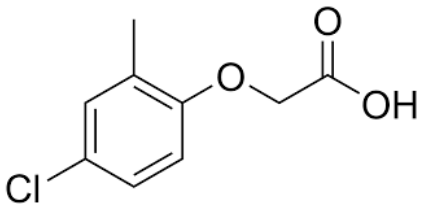
Pesticides Sectional Committee, FAD 01

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Divisional Council.

MCPA is a hormone-type herbicide readily absorbed by leaves and roots and translocated, and used for the control of annual and perennial weeds in cereals, grassland and turf.

MCPA is the accepted common name by the International Organization for Standardization (ISO) for 4-chloro-2-methylphenoxyacetic acid, also known as 4-chloro-O-cresoxyacetic acid and MCP. The empirical and structural formula and the molecular mass of MCPA are as indicated below:

Empirical Formula	*Structural Formula*	*Molecular Weight*
$C_9H_9ClO_3$		200.5

This standard was first published in 1977. In this revision, the standard has been brought into latest style and format of Indian Standards, and references to Indian Standards wherever applicable have been updated.

This standard was first In the preparation of this standard, due consideration bas been given to the provisions of the *Insecticides Act*, 1968, and the rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

The composition of the committee responsible for the formulation of this standard is listed in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022. 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

MCPA, TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for MCPA, technical.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

IS No.	*Title*
IS 460 (Part 1) : 2020	Test sieves — Specification: Part 1 Wire cloth test sieves (*fourth revision*)
IS 1070 : 1992	Reagent grade water (*third revision*)
IS 8190 (Part 1) : 1988	Requirement for packing of pesticides: Part 1 Solid pesticides (*second revision*)
IS 8294 : 2023	Liquid amine salts of MCPA — Specification (*first revision*)
IS 10946 : 1996	Methods of sampling for technical grade pesticides

3 REQUIREMENTS

3.1 Description

The material shall essentially comprise 4-chloro-2-methylphenoxyacetic acid and shall be in the form of white to brown, granular solid, free-flowing powder with a slight odour.

3.2 The material shall also comply with the requirements specified in Table 1.

4 PACKING

The material shall be packed in clean and dry containers made of mild steel. For packs up to 10 kg, tinplate containers may be used. The containers shall also comply with the general requirements as stipulated in **2** of IS 8190 (Part 1).

5 MARKING

5.1 The containers shall be securely closed and shall bear legibly and indelibly the following information and any other information as necessary under the *Insecticides Act*, and rules:

a) Name of the material;
b) Name and address of the manufacturer;
c) Date of manufacture;
d) Date of expiry;
e) Batch number;
f) Net quantity;
g) Nominal MCPA content, including the extractable acids, percent (*m/m*); and
h) Cautionary notice as worded in the *Insecticides Act*, 1968, and Rules framed thereunder; and
j) Any other information required under the *Legal Metrology* (*Packaged Commodities*) *Rules*, 2011.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn in accordance with IS 10946.

7 TESTS

7.1 Tests shall be carried out by the appropriate methods referred to in co1 (4) of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — ‘Pure chemicals l shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for MCPA, Technical

(*Clause* 3.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Extractable acids including MCPA (expressed as 4-chloro-2-methylphenoxy acetic acid),percent by mass, *Min*	94	Annex A of IS 8294
ii)	MCPA, percent by mass, *Min*	70	Annex A
iii)	Free phenols (expressed as 4-chloro-2-methyl-phenol), percent by mass, *Max*	1.5	Annex B of IS 8294
iv)	Material insoluble in NaOH, percent by mass, *Max*	0.05	Annex B
v)	Sulphate ash, percent by mass, *Max*	1.0	Annex C

ANNEX A

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF MCPA CONTENT

A-1 PRINCIPLE

MCPA (4-chloro-2-methylphenoxyacetic acid) is separated from associated acids by partition chromatography on a celite column, and is determined by titration with sodium hydroxide.

A-2 APPARATUS

A-2.1 Chromatographic Column — 65 cm long, internal diameter 10 mm to 11 mm, fitted at the lower end with a stop-cock and at the upper end with a ground glass socket to take a separating funnel.

A-2.2 Separating Funnel – 500 ml capacity, to fit the column.

A-3 REAGENTS

A-3.1 Phosphate Buffer — *p*H 6.6. Prepared by dissolving 34 g potassium dihydrogen orthophosphate in about 50 ml water. Add 39 ml of sodium hydroxide solution (1 N) and 300 ml water. Check the *p*H of the solution, and adjust, if necessary, with more sodium hydroxide, Make up to one litre with water.

A-3.2 Diethyl Ether

A-3.3 Cyclohexane

A-3.4 Tertiary Amyl Alcohol

A-3.5 Eluents

A-3.5.1 *Diethyl Ether* — Cyclohexane Mixture (A) – 1 + 1 by volume Equilibrate the mixture (1 litre) by shaking with 25 ml phosphate buffer in a separating funnel. Filter after equilibrium to remove suspended droplets.

A-3.5.2 *Diethyl Ether (B)* — Equilibrate as in **B-3.5.1**. The aqueous phase is used for buffering the support

A-3.5.3 *Diethyl Ether* — Amy/Alcohol (C) — 95 : 5 by volume. Equilibrate as in **B-3.5**.

A-3.5.4 *Diethyl Ether* — Amyl Alcohol (D) — 92 : 8 by volume. Equilibrate as in **B-3.5.1**

A-3.6 Bromothymol Blue — 0.04 percent, neutral aqueous solution

A-3.7 Standard Sodium Hydroxide Solution — 0.005N

A-3.8 Silica Gel — of such fineness that it passes through a 125 micron IS Sieve but is retained on 75 micron IS Sieve [*see* IS 460 (Part 1)]. Heat with 5 N hydrochloric acid on a boiling water-bath for 10 h, and then wash with distilled water until free from chloride ions. Dry at 140 °C for 4 h

A-4 PROCEDURE

A-4.1 Preparation of the Column — Mix well, 20 g of the prepared silica gel in small portions with the buffer solution, and make into a paste by small additions of cluent A. Plug the bottom of the column with a small plug of glass wool, half fill the tube with eluent A, and then add the prepared paste in small portions, carrying down the decanted material with the packing disc. After each addition apply gas pressure to the top of the column to assist in the removal of the solvent.

The silica gel shall always remain covered with eluent. In case, level of eluent falls below the level of the silica gel, the column shall be changed. The total length of the finished silica gel column should be between 55 cm and 60 cm. Cover the top of the silica gel with a circle of filter paper.

A-4.2 Determination of MCPA

A-4.2.1 Weigh, accurately, sufficient quantity of the sample to contain about 0.7 g of MCPA. Dissolve in eluent A in the volumetric flask, and then make up to 100 ml (solution M).

A-4.2.2 Allow the level of the liquid in the column to fall to about 2 mm or 3 mm above the silica gel. Add, from the pipette, delivering between two graduations, 1 ml of solution M. Again allow the level to fall to 2 mm or 3 mm above the silica gel, wash the inner walls of the column with 1 ml of eluent A; and repeat with a further 1 ml portion. Put 125 ml cluent A into the top funnel, and assemble

the apparatus. Eluate, until the first acid (4,6-dichloro-2-methylphenoxyacetic acid) has been eluated, collecting the eluate in 4 ml to 5 ml fractions in the conical flasks, which contain 4 ml water coloured blue by 3 drops of bromothymol blue. The rate of eluation should be about 45 ml per hour. After the first acid has been eluated collect three blank fractions and if any eluent A remains in the separating funnel, detach the funnel, and remove surplus eluent A from the column. When not more than 2 mm of eluent A remains above the silica gel, attach a separating funnel containing 100 ml of eluent B and continue the eluation until all the 4-chloro-2-methylphenoxyacetic acid has left the column. Remove the funnel of eluant B and any surplus eluent B as before, replace with a separating funneI containing 50 ml of eluent C and eluate until all the third acid (6-chloro-2-methylphenoxyacetic acid) has been eluated. Finish the eluation with 100 ml of eluent D.

A-4.2.3 As soon as it has been collected, titrate each fraction with the standard sodium hydroxide solution, to a clear blue end point. During the titration pass a current of air (free from carbon dioxide) or nitrogen through the liquid, and stir the liquid magnetically to ensure thorough mixing of the two phases.

NOTE — The first fraction needs only a drop of standard sodium hydroxide solution. The arrival of the 4, 6-dichloro-2-methylphenoxyacetic acid from the 4- chloro-2-methylphenoxyacetic acid at the bottom of the column is shown by the increased volumes of sodium hydroxide required, which rise to a peak and then die away to a minimum marking the separation of the 4, 6-dichloro-2-methylphenoxyacetic acid from the 4-chloro-2-methylphenoxyacetic acid. There may be two peaks for the 4-chloro-2-methylphrnoxyacetic acid, the second lagging behind owing to the late arrival of eluent B. Similarly, for 6-chloro-2-methylphenoxyacetic acid and, finally, for the 2-methylphenoxyacetic acid.

A-4.2.4 Construct the chromatogram by plotting the number of the fractions along the abscissa and the volumes of the sodium hydroxide used along the ordinate. Determine the quantity of standard sodium hydroxide solution corresponding to each acid. A base line correction should be made while taking the sum of the titres.

A-4.3 Calculations

$$\text{MCPA, percent by mass} = \frac{(2.006\ VN)}{M}$$

where

V = volume, in ml, of the standard sodium hydroxide solution corresponding to 4-chloro -2-methylphenoxyacetic acid;

N = normality of standard sodium hydroxide solution; and

M = mass, in g, of the sample taken for the test.

ANNEX B

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF MATERIAL INSOLUBLE IN SODIUM HYDROXIDE

B-1 REAGENT

B-1.1 Sodium Hydroxide Solution — 0.05 N

B-2 PROCEDURE

B-2.1 Weigh accurately 1 g of the sample and transfer to a 30 ml stoppered test tube. Dissolve the material using 10 ml sodium hydroxide solution, by shaking. Examine the resulting solution to *see* whether it is free from appreciable sediment. Shake the solution and pour it through a 150 micron IS Sieve [*see* IS 460 (Part 1)], washing out any residue from the test tube with water on to the sieve. Wash the residue on the sieve several times with distilled water and allow to drain. Brush the residue remaining on the sieve on to the glazed paper and then to the tared weighing bottle. Dry the weighing bottle and residue at 100 °C, cool and reweigh. Calculate the percent by mass of material insoluble in sodium hydroxide.

ANNEX C

[*Table* 1*, Sl No.* (v)]

DETERMINATION OF SULPHATED ASH

C-1 PROCEDURE

C-1.1 Weigh accurately about 5 g of the sample into a tared silica crucible with minimum dimensions of 51 mm × 51 mm. Moisten well with 95 percent ethanol (or industrial methylated spirit) and add 5 drops of concentrated sulphuric acid. Evaporate to dryness over a period of 1 h to 2 h by heating gently on a gauze or asbestos board, avoiding sputtering. Ignite until most of the carbon is burnt off. Allow to cool, add a few more drops of concentrated sulphuric acid, and re-ignite to constant mass.

C-2 CALCULATION

C-2.1 Sulphated ash, percent by mass = $\frac{100m}{M}$

where

m = mass, in g, of the sulphated ash; and
M = mass, in g, of the material taken for the test.

ANNEX D

(*Foreword*)

COMMITTEE COMPOSITION

Pesticides Sectional Committee, FAD 01

Organization	*Representative*(*s*)
Directorate of Plant Protection Quarantine and Storage, Faridabad	DR RAVI PRAKASH **(*Chairperson*)**
All India Biotech Association, New Delhi	SHRI SAURABH SINGHAL SHRI SHAH JI DHAR (*Alternate*)
Central Insecticide Board and Registration Committee, Faridabad	SECRETARY DR VANDANA SETH (*Alternate*)
Central Insecticide Laboratory, Faridabad	DR ARCHANA SINHA SHRI SUBHASH CHAUDHARY (*Alternate*)
Consumer Guidance Society of India, Mumbai	SHRI SITARAM DIXIT DR M. S. KAMATH (*Alternate*)
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Crop Life India, New Delhi	SHRI ASITAVA SEN MS NIRUPAMA SHARMA (*Alternate*)
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Ministry of Agriculture, Department of Agriculture, Chennai	JOINT DIRECTOR OF AGRICULTURE (RES) DEPUTY DIRECTOR LAB (*Alternate*)
National Centre for Integrated Pest Management, New Delhi	DR SUMITRA ARORA
National Institute of Plant Health Management, Hyderabad	DR MAHESH SAINI MS T. SRIDEVI (*Alternate*)

Organization	*Representative*(*s*)
Regional Pesticides Testing Laboratory, Chandigarh	SHRI V. VASU
In Personal Capacity (*4-6-90/2/8/2, Sri Devi Nilayam Tejaswinagar Coloni, Attapur – 500048*)	SHRI C. V. RAO
In Personal Capacity (*263, Sector 28, Faridabad – 121008*)	SHRI VIPIN SAINI
BIS Directorate General	SHRIMATI SUNEETI TOTEJA, SCIENTIST 'E'/DIRECTOR AND HEAD (FOOD AND AGRICULTURE) [REPRESENTING DIRECTOR GENERAL (*Ex- officio*)]

Member Secretary
SHRI KULDEEP MITTAL
SCIENTIST 'B'/ASSISTANT DIRECTOR
(FOOD AND AGRICULTURE), BIS

Bureau of Indian Standards

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Review of Indian Standards

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This Indian Standard has been developed from Doc No.: FAD 01 (20227).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	2323 7617
Eastern	: 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	2367 0012 2320 9474
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	265 9930
Southern	: C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	2254 1442 2254 1216
Western	: Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093	2821 8093

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Published by BIS, New Delhi